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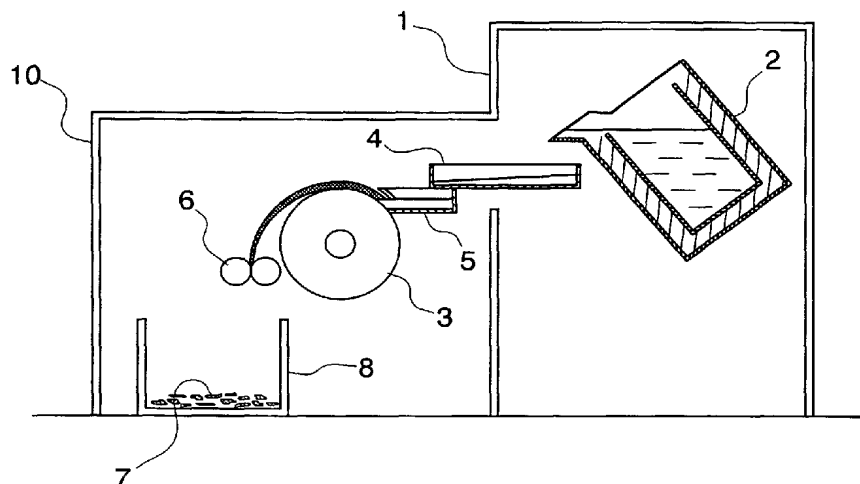
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(54) Title: METHOD FOR CONTROLLING STRUCTURE OF RARE EARTH ELEMENT-CONTAINING ALLOY, POWDER MATERIAL OF THE ALLOY AND MAGNET USING THE SAME



(57) Abstract: An object of the present invention is to provide a method for controlling an internal metallographic structure of an alloy, particularly the distribution of R-rich phase; in order to achieve the object, the present invention provide a method for controlling the metallographic structure of an alloy containing a rare earth element, which comprises the steps of: melting said alloy containing a rare earth element in vacuum or under an inert gas; pouring a molten metal obtained by melting said alloy onto a rotatable roll which is rotating and being cooled, to thereby cool the molten metal for solidification, said pouring being performed in a vacuum chamber or a chamber purged by an inert gas; forming a strip of said metal through solidification; immediately after solidification, reducing said resultant solidified strip into flakes; collecting said resultant fragmented alloy flakes in a receptacle; and controlling the cooling rate of said fragmented alloy flakes by means of a cooling medium.



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## DESCRIPTION

METHOD FOR CONTROLLING STRUCTURE OF RARE EARTH  
ELEMENT-CONTAINING ALLOY, POWDER MATERIAL OF THE ALLOY AND  
MAGNET USING THE SAME

## TECHNICAL FIELD

The present invention relates to a method for controlling an internal metallographic structure of an alloy containing a rare earth element, and more particularly to a method for controlling an internal metallographic structure of an alloy containing a rare earth element for producing magnets. The invention also relates to an alloy powder obtained from the method and to a magnet produced from the powder.

## BACKGROUND ART

In recent years, production of Nd-Fe-B alloys serving as magnet alloys has sharply increased by virtue of high-performance characteristics of the alloys, and these alloys are employed in HDs (hard disks), MRI (magnetic resonance imaging), a variety of motors, etc. Typically, a portion of Nd atoms is substituted by another rare earth element such as Pr or Dy (as used herein, Nd and the substituted Nd are referred to as R) and a portion of Fe is substituted by another transition metal element such as Co or Ni (as used herein, Fe and the substituted Fe are referred to as T). Such substituted alloys as well as Nd-Fe-B alloys are generally referred to as R-T-B alloys.

An R-T-B alloy contains, as the main phase, a ferromagnetic phase formed of  $R_2T_{14}B$  crystals, which contribute to magnetization, and, in grain boundaries of the  $R_2T_{14}B$  crystals, a nonmagnetic R-rich phase having a low melting point and containing a rare earth element(s) at high concentration. The R-T-B alloy is an active metallic material. Therefore, the alloy is generally melted and mold-cast in vacuum or under inert gas.

In a typical method of producing a magnet, an ingot of the alloy is pulverized to powder having a particle size of about 3  $\mu m$  (as measured by means of FSSS (Fisher Sub-Sieve Sizer)); the powder is subjected to press-forming in a magnetic field; the resultant compact is sintered in a sintering furnace at a temperature as high as approximately 1,000-1,100°C; and in accordance with needs, the sintered product is heated, mechanically processed, and plated for corrosion prevention.

The R-rich phase plays the following important roles in the alloy.

- (1) Since the R-rich phase has a low melting point, the phase liquefies during sintering, thereby contributing to achievement of high density of the resultant magnet, leading to improved magnetization.
- (2) The R-rich phase functions to smoothen grain boundaries, thereby reducing the number of nucleation sites in a reversed magnetic domain, thereby enhancing the coercivity.
- (3) The R-rich phase magnetically insulates the main phase, thereby enhancing the coercivity.

Thus, attainment of a uniformly dispersed R-rich phase is critical, because otherwise magnet characteristics and corrosion resistance of the produced magnet are adversely affected.

The distribution of the R-rich phase in a magnet—the final product—depends greatly on the metallographic structure of the raw material alloy ingot. Specifically, when the alloy is mold-cast, a slow cooling rate often results in formation of large crystal grains. In such a case, the particle size of the pulverized product becomes considerably smaller than that of the crystal grain size. Since the R-rich phase has a lamellar structure having thick sub-layers, the dispersibility of the phase is poor. If the particle size of the pulverized product is smaller than that of the crystal grain size, particles formed only of the main phase containing no R-rich phase and those formed only of the R-rich phase result, making it difficult to mix the main phase and R-rich phase homogeneously.

Another problem involved in mold casting is that  $\gamma$ -Fe tends to be formed as primary crystals, due to the slow cooling rate. At approximately 910°C or lower,  $\gamma$ -Fe transforms into  $\alpha$ -Fe, which deteriorates pulverization efficiency during production of magnets. If  $\alpha$ -Fe remains even after sintering, magnetic properties of the sintered product is deteriorated. Thus, the ingot obtained through mold casting must be subjected to homogenization treatment at high temperature for a long period of time in order to remove  $\alpha$ -Fe.

In order to solve the above problems, the strip casting method (SC method), which ensures a cooling rate during casting faster than that attainable by mold casting, is proposed and employed in actual production steps.

In strip casting, a molten metal is poured onto a copper roll of which inside is

cooled by water, and is formed into a strip having a thickness of some tenth mm. During casting, the molten metal was solidified through rapid cooling, to thereby yield an alloy having a microcrystalline structure in which R-rich phase is minutely dispersed. Since the R-rich phase is minutely dispersed in the alloy, dispersion of R-rich phase in the product obtained by pulverizing and sintering the alloy becomes also satisfactory, to thereby successfully attain improved magnetic characteristics (Japanese Patent Application Laid-Open (*kokai*) Nos. 5-222488 and 5-295490). In addition, generation of  $\alpha$ -Fe is suppressed.

Typically, the alloy cast through the SC method is disintegrated through hydrogen decrepitation. The SC method is based on the following specific phenomenon: when hydrogen is incorporated into the R-rich phase, the volume of the alloy ingot expands, leading to crack generation originating from the R-rich phase and disintegration of the ingot into pieces. Since hydrogen decrepitation is carried out before micro-pulverization, control of inter R-rich phase spacing is an important point for determining the size of the particle to be produced.

Thus, when the distribution profile of the R-rich phase (or inter R-rich phase spacing) which greatly affects magnet characteristics is controlled, control of cooling rate during casting, particularly control of temperature near the solidification temperature of R-rich phase is important.

Japanese Patent Application Laid-Open (*kokai*) No. 8-176755 discloses that the R-rich phase (called "eutectic crystal domain" in the disclosure) is present in the main phase ( $R_2T_{14}B$  phase) as well as in grain boundaries; that control of inter R-rich phase spacing is important for determining magnet characteristics; and that the cooling rate within a temperature range (800-600°C) where a portion of the R-rich phase that remains as a liquid phase is completely solidified is preferably controlled to 5°C/sec or more.

Japanese Patent Application Laid-Open (*kokai*) No. 10-36949 discloses that the average cooling rate within 800-600°C is controlled to be 1.0°C/sec or less, to thereby increase inter R-rich phase spacing to 3-15  $\mu\text{m}$ .

As described above, since control of the distribution profile of the R-rich phase in an alloy ingot is critical for determining magnet characteristics, the cooling rate within a temperature range of from the liquidus temperature to the solidification temperature of liquid R-rich phase must be controlled. In the aforementioned SC method, the liquidus temperature of the temperature range corresponds to the temperature of the alloy

immediately before falling from the roll, and solidification of the alloy is incomplete even after falling from the roll. Currently, no definite method has been disclosed for controlling the related temperature. Regulation of the cooling rate of a metallic material present on the roll has heretofore been attained either by modification of the peripheral velocity of the roll or by adjustment of the flow of the metal for modifying the thickness of the metal layer. These methods involve problems which are difficult to be solved. Specifically, when solidification of the main phase is complete, the manner of contact between the metal and the roll is changed from face-to-face to point-to-face, to thereby suddenly retard the cooling rate. If an alloy ingot of favorable metallographic structure, having no  $\alpha$ -Fe, is to be produced smoothly, molten metal and an alloy ingot in which the main phase is solidified are in contact with the roll for only few seconds. In such a short period of time, the temperature range in which complete solidification of R-rich phase on the roll is attained cannot be controlled. When the period of time during which the alloy ingot is in contact with the roll is prolonged by decreasing the peripheral velocity of the roll, the thickness of the alloy ingot increases, generating  $\alpha$ -Fe. When the amount of the molten alloy to be supplied onto the roll is reduced by slowing the tilting speed of the crucible, the temperature of the molten alloy decreases before it reaches the roll, causing easy generation of  $\gamma$ -Fe primary crystals. In addition, further reduction of the amount of the molten alloy to be supplied causes solidification of the molten alloy before it reaches the roll.

Thus, when the SC method is employed, there have been no effective means for controlling, at temperatures near the solidification temperature, the cooling rate of the R-rich phase on the roll to the effect of satisfactorily changing the as-cast metallographic structure. Furthermore, there has heretofore been disclosed substantially no specific means for controlling metallographic structure of a cast alloy which has already fallen from the roll.

Accordingly, an object of the present invention is to provide a method for controlling an internal metallographic structure of an alloy, particularly the distribution of R-rich phase, by controlling the cooling rate of alloy flakes which have already fallen from the roll during casting through a conventional roll casting method using a rotating roll.

#### DISCLOSURE OF INVENTION

The present invention has been accomplished in order to attain the aforementioned object, and the invention provides a method for controlling the metallographic structure of an alloy containing a rare earth element, which comprises the steps of: melting an alloy containing a rare earth element in vacuum or under an inert gas; pouring a molten metal obtained by melting the alloy onto a rotatable roll which is rotating and being cooled, to thereby cool the molten metal for solidification, the pouring being performed in a vacuum chamber or a chamber purged by an inert gas; forming a strip of the metal through solidification; immediately after solidification, reducing the resultant solidified strip into flakes; collecting the resultant fragmented alloy flakes in a receptacle; and controlling the cooling rate of the fragmented alloy flakes by means of a cooling medium.

In the method, it is preferable for the receptacle to have a cooling partition and a gas or liquid serving as a cooling medium to be allowed to pass through the cooling partition, to thereby control the cooling rate of the fragmented alloy flakes.

In the method, it is also preferable for the receptacle to be capable of controlling the cooling rate of the fragmented alloy flakes when an inert gas serving as a cooling medium is allowed to pass through the interior of the receptacle.

In the method, it is also preferable for the inert gas to be caused to flow through the cooling partition provided in the receptacle and having a vent.

In the method, it is also preferable for the receptacle to have a separation partition for controlling the cooling rate of the fragmented alloy flakes.

In the method, it is also preferable to further comprise a step in that subsequent to controlling of the cooling rate of the fragmented alloy flakes, transferring the fragmented alloy flakes from the chamber to another chamber for cooling the fragmented alloy flakes.

In the method, it is also preferable to employ a strip casting method for pouring a molten alloy containing a rare earth element onto a rotatable roll which is rotating, to thereby cool the molten alloy and form a strip through solidification.

In the method, it is also preferable to control the cooling rate of the fragmented alloy flakes, to thereby control average inter R-rich phase spacing to 3-15  $\mu\text{m}$ .

In the method, it is also preferable for the average cooling rate of the fragmented alloy flakes to be within a range of 800 to 600°C to be 10-300°C/min.

Moreover, in the method, it is also preferable for the alloy containing a rare earth element to be an R-T-B alloy, wherein R represents at least one element selected from

among rare earth elements including Y; that is, at least one element selected from the group consisting of Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu; and T represents a substance predominantly comprising Fe, with a portion of Fe atoms being optionally substituted by Co, Ni, etc.

In addition, the present invention has been accomplished in order to attain the aforementioned object, and the invention provides a powder of an alloy containing a rare earth element, which powder is produced by pulverizing fragmented alloy flakes obtained by the method and having a thickness of 0.1-0.6 mm and an inter R-rich phase spacing of 3-15  $\mu\text{m}$ .

Furthermore, the present invention has been accomplished in order to attain the aforementioned object, and the invention provides a magnet produced by shaping a powder of the alloy and sintering the shaped product.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic view depicting one method for melting and casting an alloy containing a rare earth element.

FIG. 2 is a sketch depicting one method for cooling flakes of a cast and fragmented alloy contained in a receptacle.

FIG. 3 is a sketch depicting another cooling method.

FIG. 4 is a sketch depicting another cooling method.

FIG. 5 is a sketch depicting another cooling method.

FIG. 6 is a sketch depicting another cooling method.

#### MODES FOR CARRYING OUT THE INVENTION

Generally, when an alloy containing a rare earth element is cast, the alloy is molten and, due to its active property, is cast in an air-tight melting chamber. The interior of the melting chamber is controlled to be vacuum or an inert gas (e.g., argon or helium) atmosphere. FIG. 1 is a schematic view showing one casting method based on the SC method employed according to the present invention. Metallic raw materials are molten in a crucible 2 placed in a melting chamber 1 through induction heating, to thereby yield an alloy which is then maintained in the crucible. The thus-produced molten alloy is transferred, by tilting the crucible 2, to a trough 4, and then to a tundish 5. The molten alloy is poured onto a rotatable roll 3 which is rotating and placed in a vacuum or an inert



gas casting chamber 10 adjacent to the melting chamber. The interior of the roll is cooled with water, and the alloy begins to cool and solidify on the roll 3.

The thus-solidified alloy is released from the roll 3 as the roll rotates. The released alloy is forced to fall by use of an appropriate guide, so as to prevent re-contact of the alloy with the roll 3.

The alloy as released from the roll is fragile due to its high temperature, and is readily fractured by means of a braking means such as simple guide rolls 6 or, in an easier manner, by bringing into contact with an obstacle such as a baffle plate. Thus, the alloy is reduced into flakes, and the flakes fall. Alternatively, the alloy may be fragmented by impact upon falling into a receptacle 8.

The period of time from the contact of the molten alloy with the roll to reduce the alloy in size released from the roll is as short as some seconds. As described above, the solidified alloy at this stage is still in a glowing state, and the R-rich phase having the lowest solidification temperature has not been completely solidified.

Typically, the fragmented alloy flakes 7 are forced to fall into the box-like receptacle 8 placed adjacent to the roll and are collected therein. Since oxidation of the alloy readily proceeds at high temperature, the receptacle is typically placed in the casting chamber 10, also accommodating the casting apparatus, and the alloy is cooled to a temperature at which oxidation of the collected alloy does not occur. Japanese Patent Application Laid-Open (*kokai*) No. 9-155507 discloses that a receptacle in which fragmented alloy flakes are placed is transferred into another chamber adjacent to the receptacle, and the flakes are cooled by use of a medium such as an inert gas. However, the cooling step is not intended to control the metallographic structure of the alloy.

The present invention is firstly drawn to a method for controlling the cooling temperature of alloy flakes, and the method includes placing a receptacle for accommodating fragmented cast alloy flakes (hereinafter referred to as alloy flakes) in order to control the metallographic structure at the solidification temperature, particularly control the R-rich phase of low-melting point; and controlling the cooling temperature of the alloy flakes in the receptacle by means of a cooling medium while the alloy flakes in the receptacle. This method enables cooling of alloy flakes while they are falling into a receptacle. Thus, as compared with the method disclosed in the aforementioned patent publication in which a receptacle is transferred into another chamber for cooling after completion of casting, the method of the present invention attains uniform cooling, and

cooling rate can be controlled within a wide temperature range by virtue of small decrease in temperature before the start of control of the cooling rate. In addition, if necessary, when the receptacle is not cooled, it is possible to maintain the temperature of the alloy due to only heat of the alloy itself. According to the present method, the cooling rate within a temperature range of 800-600°C can be controlled easily; note that the cooling rate within this range significantly affects the metallographic structure of alloy.

Secondly, in view that the cooling rate after completion of solidification of R-rich phase and the internal metallographic structure has become solid does not affect the internal metallographic structure, alloy flakes must be removed as quickly as possible so as to meet the process requirement. Thus, cooling to about 100-200°C, where oxidation does not proceed and alloy flakes can be removed into the air, is preferably performed as rapidly as possible under an atmosphere such as inert gas.

In order to meet the above two requirements, for example, a receptacle shown in FIG. 2 is employed. Specifically, a stainless-steel net 233 is provided in the bottom section of the receptacle, and an inert cooling gas 23 such as helium is allowed to pass through the net. Immediately after the falling alloy flakes have been collected, gas is allowed to pass. The cooling rate of the alloy flakes can be altered by changing the flow rate of the gas. When the temperature of the alloy flakes has reached a temperature lower than the aforementioned R-rich phase solidification temperature range; i.e., 800-600°C, cooling can be performed at a maximum gas flow rate until the temperature has reached a point where alloy flakes can be removed to the air.

In the above embodiment, cooling is performed through contact of deposited alloy flakes with a gas which is allowed to pass through between the deposits. Thus, when a large amount of alloy flakes are deposited and a receptacle having a large volume is employed, control of the cooling rate is sometimes restricted, or difference in cooling effect readily occurs in the alloy flakes placed in the receptacle.

In such a case, as shown in FIG. 3, the inside of the receptacle is separated by providing hollow partitions 211, and a cooling medium 22 is introduced in each partition, to thereby enhance the cooling rate alloy flakes through contact between the partition and the flakes. In this method, in addition to inert gas, a gas such as air or a liquid such as water can be used as a cooling medium, since the cooling medium is not in contact with the alloy flakes.

Alternatively, a cooling method employing a receptacle shown in FIG. 4 may also

be applicable. FIG. 4 shows a cooling method in which an inert gas 23 for cooling alloy flakes is introduced from a vent 212A provided in the bottom of a cooling partition 212 and a portion of the gas is allowed to flow through the receptacle, to thereby cool alloy flakes.

After the internal metallographic structure of the alloy has become solidified, subsequent cooling is performed as rapidly as possible to attain high cooling efficiency. Particularly when casting is performed successively, such rapid cooling is preferred. Rapid cooling may be performed in a casting chamber as described above. Alternatively, rapid cooling may be performed in another chamber into which the receptacle is transferred.

When the receptacle is removed from the casting chamber and transferred to another chamber charged with an inert gas, the top of the receptacle may be covered with a lid, and the receptacle can be cooled again in the chamber. Complete sealing of the receptacle is not needed during transfer, and an inert gas may be supplied continuously to the receptacle during transfer in an amount such that the gas overflows from the receptacle. If the time spent for transferring is short, gas supply may be stopped, with the top of the receptacle remaining lidded, after completion of charging the gas. In this case, a hose, etc. for supplying the gas is removed from the receptacle, and the connection portion is plugged. Because an inert gas such as argon is heavier than the air, the gas does not leak from the receptacle even when the receptacle is not completely lidded.

In another method for causing an inert gas to flow, the gas may be supplied from vents 213A which are provided on the hollow partition 213 side, as shown in FIG. 5.

Each of the receptacles shown in FIGs. 5 and 6 is provided in the center thereof with partitions 24 for separating the inside thereof. In these receptacles, alloy flakes are divided into small portions, and therefore, readily cooled. If these separating partitions are not provided, alloy flakes are localized in the receptacle, to thereby form aggregates thereof, and cooling may be hampered or easily melt and adhered in an aggregated state. Cooling is performed by supplying an inert gas into the receptacle from vents 213A provided in each cooling partition 213 or a stainless-steel net 233 provided in the bottom section of the receptacle. A cooling method employing a receptacle shown in FIG. 3 or 4 may also be employed. After completion of cooling, the receptacle is removed through, for example, an openable, closable door which is provided in a side face of the casting chamber.

According to the aforementioned two-step method for controlling the cooling rate within the receptacle, distribution of R-rich phase can be controlled by controlling the temperature particularly within the first high temperature range. Regardless of the cooling rate within the first high temperature range, cooling within the second temperature range where no effect is caused on the internal metallographic structure can be performed rapidly, to thereby realize a smooth progress of the steps.

When alloy flakes having a thickness of 0.1-0.6 mm produced by casting through the method, i.e., SC method, the flakes which have been released from the roll and fallen into a receptacle have a temperature of approximately 800°C. By employing any mode selected from the aforementioned cooling methods performed in the receptacle, the cooling rate within the first temperature range is controlled to be slow, to thereby increase inter R-rich phase spacing, and the cooling is controlled to be fast, to thereby decrease inter R-rich phase spacing. In the present invention, the casting method is not limited to an SC method as shown in FIG. 1, and other methods, such as casting by pouring a molten alloy into the space between rotatable twin rolls which are rolling may also be employed.

According to the method of the present invention, inter R-rich phase spacing of an alloy containing a rare earth element such as R-T-B alloy (R represents at least one element selected from among rare earth elements including Y; that is, at least one element selected from the group consisting of Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu; and T represents a substance predominantly comprising Fe, with a portion of Fe atoms being optionally substituted by Co, Ni, etc.) can be controlled to be approximately 3-15  $\mu\text{m}$ . The average cooling rate within the range of 800-600°C for controlling inter R-rich phase spacing to fall within the above range is suitably 10-300°C/min, preferably 10-200°C/min, more preferably 10-50°C/min.

Inter R-rich phase spacing is obtained through the following procedure. Specifically, a cross-section, as cut in the thickness direction, of an alloy flake is polished by use of an emery sheet and buffed by use of alumina, diamond, etc., and the thus-polished surface is observed under a scanning electron microscope (SEM), to thereby obtain a reflected electron image. In the image, the R-rich phase is observed as a brighter region, since atoms present in the R-rich phase have an average atomic number higher than that of the atoms present in  $\text{R}_2\text{Fe}_{14}\text{B}$  phase serving as the main phase. Inter R-rich phase spacing is measured through observation of the cross-section in the thickness direction. More specifically, a line segment parallel to a roll surface (in a direction

parallel to the roll shaft) is drawn in the middle of the thickness-direction cross-section. The length of the line segment is divided by the number of times the R-rich phase traverses the line segment. The observation is repeated five times for different observation areas. The average value serves as inter R-rich phase spacing.

According to the present invention, inter R-rich phase spacing can be controlled to 3-15  $\mu\text{m}$ , preferably 3-12  $\mu\text{m}$ , more preferably 4-10  $\mu\text{m}$ . In the case in which this is in excess of 15  $\mu\text{m}$ , dispersion of R-rich phase is unsatisfactorily attained, and the proportion of powder particles including R-rich phase decreases when the powder particles having a particle size of 3-5  $\mu\text{m}$  for shaping in magnetic field are formed through micro-pulverization. Thus, dispersion of R-rich phase of a product shaped in magnetic field is deteriorated, leading to lowering in sinterability and lowering in magnetization and coercive force of produced magnets. In addition, localization of R-rich phase results in local lowering in coercive force, leading to lowering in squareness ratio of produced magnets. In contrast, when the inter R-rich phase spacing is less than 3  $\mu\text{m}$ , crystal grains are excessively micro-pulverized, disadvantageously leading to deterioration in magnetic characteristics.

Subsequently, the aforementioned alloy flakes are pulverized, shaped, and sintered, to thereby produce anisotropic magnets of excellent characteristics.

Typically, pulverization is sequentially performed in the order of hydrogen decrepitation, intermediate pulverization, and micro-pulverization, to thereby produce a powder generally having a size of approximately 3-5  $\mu\text{m}$  (FSSS).

In the present invention, hydrogen decrepitation includes a hydrogen absorption step as a first step and a hydrogen desorption step as a second step. In the hydrogen absorption step, hydrogen is caused to be absorbed predominantly in the R-rich phase of alloy flakes in a hydrogen gas atmosphere at  $2.7 \times 10^4$  Pa to  $4.9 \times 10^6$  Pa. The R-rich phase is expanded in volume due to R hydride generated in this step, to thereby minutely reduce the alloy flakes themselves or generate numerous micro-cracks. In the case of the alloy flakes of the present invention, cracks can be generated along almost the entire portions of the R-rich phase. Particularly, since grain boundaries of the main phase contains an R-rich phase, absorption of hydrogen generates cracks in almost the entire portions of the grain boundaries. Accordingly, a powder produced by subjecting to intermediate pulverization and micro-pulverization is formed of mono-crystalline particles,

to thereby enhance magnetic characteristics. Hydrogen absorption is carried out within a temperature range of ambient temperature to approximately 600°C. However, in order to increase expansion in volume of R-rich phase so as to effectively reduce the flakes in size, hydrogen absorption is preferably performed within a temperature range of ambient temperature to approximately 100°C. The time for hydrogen absorption is preferably one hour or longer. The R hydride formed through the hydrogen absorption step is unstable in the atmosphere and readily oxidized. Thus, the hydrogen-absorbed product is preferably subjected to hydrogen desorption treatment by maintaining the product at 200-600°C in vacuum of 130 Pa or less. Through this treatment, R hydride can be transformed into a product stable in the atmosphere. The time for hydrogen desorption treatment is preferably 30 minutes or longer. If the atmosphere is controlled for preventing oxidation during steps to be carried out after hydrogen absorption to sintering, hydrogen desorption treatment can also be omitted.

Alternatively, pulverization may be performed through intermediate pulverization and micro-pulverization without performing hydrogen decrepitation.

Intermediate pulverization is a pulverization step in which alloy flakes are pulverized in an inert gas atmosphere such as argon gas or nitrogen gas, to a particle size of, for example, 500 µm or less. Examples of pulverizers for performing this pulverization include a Brawn mill. In the present invention, if the alloy flakes have been subjected to hydrogen decrepitation, the alloy flakes has already been reduced minutely or has included numerous minute cracks generated therein. Thus, intermediate pulverization may be omitted.

Micro-pulverization is a pulverization step for attaining a particle size of approximately 3-5 µm (FSSS). Examples of pulverizers for performing the pulverization include a jet mill. Upon micro-pulverization, the atmosphere is controlled to an inert gas atmosphere such as an argon gas atmosphere or nitrogen gas atmosphere. The inert gas may contain oxygen in an amount of 2% by mass or less, preferably 1% by mass or less. The presence of oxygen enhances pulverization efficiency and attains oxygen concentration of the powder produced through pulverization to 1,000-10,000 ppm, to thereby enhance oxidation resistance. In addition, abnormal grain growth during sintering can be prevented.

Molding is performed in a magnetic field. In order to reduce friction between the powder and the inner wall of a mold and to reduce friction generated among powder

particles for enhancing orientation, a lubricant such as zinc stearate is preferably added to the powder during molding in magnetic field. The amount the lubricant to be added is 0.01-1% by mass. Although the lubricant may be added before or after micro-pulverization, the lubricant is preferably mixed sufficiently, before molding in magnetic field, in an inert gas atmosphere such as argon gas or nitrogen gas by use of a mixing apparatus such as a V-blender.

The powder obtained through micro-pulverization is press-molded in magnetic field by use of a molding apparatus. The mold to be employed is fabricated from a magnetic material and a non-magnetic material in combination in consideration of the orientation of magnetic field in the mold cavity. The pressure at molding is preferably 0.5-2 t/cm<sup>2</sup>, and the magnetic field in the mold cavity during molding is preferably 5-20 kOe. The atmosphere during molding is preferably an inert gas atmosphere such as argon gas or nitrogen gas. However, if the powder has been subjected to the aforementioned anti-oxidation treatment, molding can be performed in air.

Sintering is performed at 1,000-1,100°C. Prior to sintering, a lubricant and hydrogen must be completely removed from a compact to be sintered. The lubricant is removed by maintaining the compact preferably under the conditions: in vacuum of 1.3 Pa or less or under an argon gas flow atmosphere at reduced pressure; at 300-500°C; and for 30 minutes or longer. Hydrogen is removed by maintaining the compact preferably under the conditions: in vacuum of 1.3 Pa or less; at 700-900°C; and for 30 minutes or longer. The atmosphere during sintering is preferably an argon gas atmosphere or a vacuum atmosphere of 1.3 Pa or less. A retention time of one hour or longer is preferred.

After completion of sintering, in order to enhance the coercive force, the sintered product may be treated at 500-650°C in accordance with needs. An argon gas atmosphere or a vacuum atmosphere is preferred, and a retention time of 30 minutes or longer is preferred.

The results of controlling the metallographic structure (R-rich phase) according to the present invention will next be described in detail by way of examples.

#### Example 1

Elemental neodymium, ferroboration, cobalt, aluminum, copper, and iron were mixed to thereby obtain the following alloy composition: Nd: 30.0% by mass; B: 1.00%

by mass; Co: 2.0% by mass; Al: 0.30% by mass; Cu: 0.10% by mass; and a balance of iron. The resulting mixture was melted in an alumina crucible in an argon gas atmosphere (1 atm) by use of a high-frequency induction melting furnace (crucible). The resulting molten alloy was poured, via a tundish, onto a copper roll having a diameter of 40 cm and rotating at a peripheral velocity of 0.97 m/sec. The total mass of the molten metal was 15 kg, and the temperature of the molten alloy at the start of casting was controlled to 1,450°C. During casting, the inside of the copper roll was cooled by water.

The alloy solidified on the copper roll was fragmented by means of guide rolls provided at a position where the alloy was released from the copper roll and fell. The fragmented alloy was collected in a box-like receptacle provided under the guide rolls. The receptacle had a length of 31 cm, a width of 21 cm, and a height of 40 cm (outer dimensions) and was made of an iron sheet having a thickness of 5 mm. In addition, as shown in FIG. 2, a stainless-steel net (scale spacing: 5 mm) was placed in the bottom section of the receptacle at a position 1 cm above the bottom plate, and the fragmented alloy flakes were collected on the net.

Until 10 minutes after completion of casting from immediately before the start of casting, argon gas continuously allowed to pass through the stainless-steel net toward the upper side at a flow rate of 30 liters/min.

Assuming that the temperature of the alloy during falling is approximately equal to that of the alloy flakes accumulated in the receptacle, the temperature of the alloy during falling was determined by measuring the temperature of alloy flakes by use of a thermocouple which was provided in the receptacle such that the thermocouple was protruded from a small hole made in a side face on the receptacle. The temperature of the alloy flakes during falling thus measure was found to be 780°C. The alloy flakes were gradually cooled, and the time required until the temperature reached 600°C was five minutes.

Ten minutes after completion of casting, the flow rate of argon gas passing through the stainless-steel net toward the upper side was increased to 100 liters/min, to thereby cool the alloy flakes. After two hours had been elapsed, the temperature of the alloy flakes was lowered to 98°C. Subsequently, the alloy flakes were removed to the air, and the mean thickness was measured by use of a micrometer, and the inter R-rich phase spacing was measured on the basis of an electron-reflection photographic image of a cross-section obtained through SEM. Table 1 shows the measurement results.



### Example 2

The same raw materials as those used in Example 1 were mixed, to thereby obtain the same composition as that of Example 1, and the resultant mixture was formed into alloy flakes by use of the same apparatus as that of Example 1. A receptacle shown in FIG. 2 was employed. From immediately before the start of casting, helium gas was introduced from the bottom of the receptacle at a flow rate of 100 liters/min and the gas was allowed to pass through the net continuously. The temperature of the alloy flakes during falling as measured by the above method was found to be 750°C. The time required for cooling the alloy flakes to 600°C was 40 seconds.

Even after completion of casting, helium gas was allowed to pass continuously so as to cool the alloy flakes, the temperature thereof was lowered to 96°C 30 minutes after completion of casting. Subsequently, the alloy flakes were removed to the air, and the mean thickness was measured by use of a micrometer, and the inter R-rich phase spacing was measured on the basis of an electron-reflection photographic image of a cross-section obtained through SEM. Table 1 shows the measurement results.

### Example 3

The same metallic raw materials as those used in Example 1 were mixed, to thereby obtain the same composition as that of Example 1, and the resultant mixture was melt under the same conditions as employed in Example 1. The resultant molten alloy was cast by use of the same copper roll as used in Example 1.

The alloy solidified on the copper roll was fragmented by means of the same guide rolls as employed Example 1. The fragmented alloy was collected in a box-like receptacle provided under the guide rolls. The receptacle had a length of 31 cm, a width of 21 cm, and a height of 40 cm (outer dimensions) and was made of an iron sheet having a thickness of 5 mm. In addition, as shown in FIG. 3, two iron-made partitions (thickness: 7 cm) 211 were placed inside the receptacle such that the two partitions were arranged at same intervals in the direction normal to the rotation axis of the copper roll. Each partition had structure in which a gas supplied into the partition does not leak out to the receptacle. From immediately before the start of casting, argon gas was continuously allowed to pass through each partition at a flow rate of 100 liters/min.

The temperature of the alloy during falling as measured by the same method as

employed in Example 1 was found to be 790°C. The alloy flakes were gradually cooled, and the time required for cooling the alloy flakes to 600°C was 7 minutes.

Even after completion of casting, argon gas was allowed to pass continuously so as to cool the alloy flakes, the temperature thereof was lowered to 106°C two hours after completion of casting. Subsequently, the alloy flakes were removed to the air, and the mean thickness was measured by use of a micrometer, and the inter R-rich phase spacing was measured on the basis of an electron-reflection photographic image of a cross-section obtained through SEM. Table 1 shows the measurement results.

#### Example 4

The same metallic raw materials as those used in Example 1 were mixed, to thereby obtain the same composition as that of Example 1, and the resultant mixture was formed into alloy flakes by use of the same apparatus as that of Example 3. A receptacle shown in FIG. 3 was employed. From immediately before the start of casting, water was continuously allowed to pass through each partition at a flow rate of 30 liters/min.

The temperature of the alloy during falling as measured by the same method as employed in Example 1 was found to be 790°C. The alloy flakes were gradually cooled, and the time required for cooling the alloy flakes to 600°C was 6 minutes.

Even after completion of casting, water was allowed to pass continuously so as to cool the alloy flakes, the temperature thereof was lowered to 98°C two hours after completion of casting. Subsequently, the alloy flakes were removed to the air, and the mean thickness was measured by use of a micrometer, and the inter R-rich phase spacing was measured on the basis of an electron-reflection photographic image of a cross-section obtained through SEM. Table 1 shows the measurement results.

#### Example 5

The same metallic raw materials as those used in Example 1 were mixed, to thereby obtain the same composition as that of Example 1, and the resultant mixture was melt under the same conditions as employed in Example 1. The resultant molten alloy was cast by use of the same copper roll as used in Example 1.

The alloy solidified on the copper roll was fragmented by means of the guide rolls as employed in Example 1. The fragmented alloy was collected in a box-like

receptacle provided under the guide rolls. The receptacle had a length of 31 cm, a width of 21 cm, and a height of 40 cm (outer dimensions) and was made of an iron sheet having a thickness of 5 mm. In addition, as shown in FIG. 4, two iron-made partitions (thickness: 7 cm) 212 having a structure in which a gas was allowed to flow inside the receptacle from the bottom section were placed inside the receptacle such that the two partitions were arranged at same intervals in the direction normal to the rotation axis of the copper roll. From immediately before the start of casting, argon gas was continuously allowed to pass from the vent 212A in each partition at a flow rate of 30 liters/min.

The temperature of the alloy during falling as measured by the same method as employed in Example 1 was found to be 780°C. The alloy flakes were gradually cooled, and the time required for cooling the alloy flakes to 600°C was 5 minutes.

Ten minutes after completion of casting, the flow rate of argon gas which was allowed to pass through each partition was increased to 100 liters/min, and the top of the receptacle was lidded. Subsequently, the receptacle was removed from the vacuum apparatus to the air, and immediately transferred to another chamber purged with argon gas. In order to reduce the oxygen concentration of the atmosphere of the chamber that had been increased by the transfer operation and to cool the alloy flakes, argon gas was continuously allowed to pass through each partition at a flow rate of 100 liters/min, even after the receptacle had been transferred to another chamber.

The temperature of the alloy flakes was 94°C two hours after completion of casting. Subsequently, the alloy flakes were removed to the air, and the mean thickness was measured by use of a micrometer, and the inter R-rich phase spacing was measured on the basis of an electron-reflection photographic image of a cross-section obtained through SEM. Table 1 shows the measurement results.

Moreover, the oxygen concentration of the alloy flakes was measured and found to be 140 ppm, which was equivalent to the oxygen concentration (130 ppm) of the alloy flakes of Example 1. This indicated that no oxidation of the alloy flakes was caused by transferring the receptacle.

#### Example 6

Elemental neodymium, elemental dysprosium, ferroboron, cobalt, aluminum, copper, and iron were mixed to thereby obtain the following alloy composition: Nd: 29.0% by mass; Dy: 3.5% by mass; B: 1.05% by mass; Co: 1.0% by mass; Al: 0.30% by mass;

Cu: 0.10% by mass; and a balance of iron. The resulting mixture was melted in an alumina crucible in an argon gas atmosphere (1 atm) by use of a high-frequency induction melting furnace. The resulting molten alloy was poured, via the same tundish as employed in Example 1, onto the copper roll employed in Example 1 and rotating at a peripheral velocity of 0.97 m/sec. The total mass of the molten metal was 15 kg, and the temperature of the molten alloy at the start of casting was controlled to 1,450°C.

The alloy solidified on the copper roll was fragmented by means of the guide rolls as employed in Example 1. The fragmented alloy was collected in a box-like receptacle provided under the guide rolls. The receptacle had a length of 31 cm, a width of 21 cm, and a height of 40 cm (outer dimensions) and was made of an iron sheet having a thickness of 5 mm. In addition, as shown in FIG. 5, two separation partitions (thickness: 2 cm) 24 were placed inside the receptacle such that the two partitions were arranged at same intervals in the direction normal to the rotation axis of the copper roll. The separation partitions, made of a refractory predominantly containing alumina, had a thermal conductivity at 1,000°C of 0.2 kcal/(mh°C) (0.23 W/m°C). In addition, one iron-made cooling partition (thickness: 3 cm) 213 was provided in the middle of two separation partitions. The cooling partition was hollow, and had a number of holes (diameter: 1 mm) 213A provided on each side face. The cooling partition had a structure in which an inert gas was allowed to be supplied through these holes into the receptacle, to thereby cool alloy flakes. From immediately before the start of casting to 10 minutes after completion of casting, argon gas was allowed to pass through each partition at a flow rate of 10 liters/min, and the alloy flakes were cooled by argon gas supplied from the holes provided in the side faces of each partition.

The temperature of the alloy during falling as measured by the same method as employed in Example 1 was found to be 690°C. The alloy flakes were gradually cooled, and the time required for cooling the alloy flakes to 600°C was 6 minutes.

Ten minutes after completion of casting, the flow rate of argon gas which was allowed to pass through each partition was increased to 50 liters/min, and the alloy flakes were cooled by argon gas flowing from holes provided in each side face of the partition. The temperature of the alloy flakes was 101°C two hours after completion of casting. Subsequently, the alloy flakes were removed to the air, and the mean thickness was measured by use of a micrometer, and the inter R-rich phase spacing was measured on the basis of an electron-reflection photographic image of a cross-section obtained through

SEM. Table 1 shows the measurement results.

#### Example 7

The same metallic raw materials as those used in Example 6 were mixed, to thereby obtain the same composition as that of Example 6, and the resultant mixture was melt under the same conditions as employed in Example 1. The resultant molten alloy was cast by use of the same copper roll as used in Example 1.

The alloy solidified on the copper roll was fragmented by means of the same guide rolls as employed Example 1. The fragmented alloy was collected in a box-like receptacle provided under the guide rolls. As shown in FIG. 6, the employed receptacle was similar to that of Example 1, further including three separation partitions (thickness: 2 cm) placed inside the receptacle such that the three partitions were arranged at same intervals in the direction normal to the rotation axis of the copper roll. The separation partitions were made of the same material that used in Example 6. From immediately before the start of casting to 10 minutes after completion of casting, argon gas was allowed to pass through the stainless-steel net provided in the bottom section of the receptacle toward the upside at a flow rate of 10 liters/min.

The temperature of the alloy during falling as measured by the same method as employed in Example 1 was found to be 690°C. The alloy flakes were gradually cooled, and the time required for cooling the alloy flakes to 600°C was 6 minutes.

From 10 minutes after completion of casting, argon was changed to helium, and the helium gas was allowed to pass through the stainless-steel net provided in the bottom section of the receptacle toward the upside at a flow rate of 100 liters/min, to thereby cool the alloy flakes. The temperature the alloy flakes was 103°C 30 minutes after completion of casting. Subsequently, the alloy flakes were removed to the air, and the mean thickness was measured by use of a micrometer, and the inter R-rich phase spacing was measured on the basis of an electron-reflection photographic image of a cross-section obtained through SEM. Table 1 shows the measurement results.

#### Comparative Example 1

The same metallic raw materials as those used in Example 1 were mixed, to thereby obtain the same composition as that of Example 1, and the resultant mixture was melt under the same conditions as employed in Example 1. The resultant molten alloy

was cast by use of the same copper roll as used in Example 1.

The alloy solidified on the copper roll was fragmented by means of the same guide rolls as employed in Example 1. The fragmented alloy was collected in a box-like receptacle provided under the guide rolls. The receptacle had a length of 31 cm, a width of 21 cm, and a height of 40 cm (outer dimensions) and was made of an iron sheet having a thickness of 5 mm. In the receptacle, a net, cooling partitions, separation partitions, etc. as used in Examples 1 to 7 were not provide, and cooling by an inert gas or a similar medium was not carried out. Thus, the cooling rate of alloy flakes contained in the receptacle was not controlled.

The temperature of the alloy during falling as measured by the same method as employed in Example 1 was found to be 790°C. The cooling rate of the alloy flakes was considerably slow, and the alloy flakes were gradually cooled, and the time required for cooling the alloy flakes to 600°C was one hour. Furthermore, as the temperature of the alloy flakes became lower, the cooling rate decreased. A very long period of time; i.e., eight hours after completion of casting, was required until the temperature of the alloy flakes reached 200°C, at which no further oxidation of the alloy proceeded in the air.

Subsequently, when the alloy flakes were removed to the air, alloy flakes were strongly melt-adhered one another, and the mean thickness was not able to be measured by use of a micrometer. The inter R-rich phase spacing was able to be measured on the basis of an electron-reflection photographic image of a cross-section obtained through SEM. Table 1 shows the measurement results.

Table 1-1 CAST CONDITIONS AND INTER R-RICH PHASE SPACING

	Components % by mass						
	Nd	Dy	B	Co	Al	Cu	Fe
Ex. 1	30.0	0	1.00	2.0	0.30	0.10	balance
Ex. 2	30.0	0	1.00	2.0	0.30	0.10	balance
Ex. 3	30.0	0	1.00	2.0	0.30	0.10	balance
Ex. 4	30.0	0	1.00	2.0	0.30	0.10	balance
Ex. 5	30.0	0	1.00	2.0	0.30	0.10	balance
Ex. 6	29.0	3.5	1.05	1.0	0.30	0.10	balance
Ex. 7	29.0	3.5	1.05	1.0	0.30	0.10	balance
Comp. Ex. 1	30.0	0	1.00	2.0	0.30	0.10	balance

Table 1-2 CAST CONDITIONS AND INTER R-RICH PHASE SPACING

	Cast conditions					Appearance	Cross-section structure
	Peripheral velocity of roll (m/sec)	Temp. of molten alloy during casting (°C)	Temp. of alloy flakes during falling (°C)	Gas supplied to receptacle	Cooling rate until 600°C (°C/min)		
Ex. 1	0.97	1,450	780	argon	36	0.332	Inter R-rich phase spacing (μm)
Ex. 2	0.97	1,450	750	helium	22.5	0.325	6.8
Ex. 3	0.97	1,450	790	argon	27	0.331	3.4
Ex. 4	0.97	1,450	790	(water)	32	0.336	7.5
Ex. 5	0.97	1,450	780	argon	36	0.330	7.0
Ex. 6	0.97	1,450	690	argon	15	0.328	6.8
Ex. 7	0.97	1,450	690	argon	15	0.326	4.7
Comp. Ex. 1	0.97	1,450	790	none	3	Un-measurable	4.6
							19.2



## Production of magnets

## Example 8

The alloy flakes produced in Example 1 were pulverized in the order of hydrogen decrepitation, intermediate pulverization, and micro-pulverization. Hydrogen absorption step—the first step of hydrogen decrepitation—was performed under the conditions: 100% hydrogen atmosphere, 1 atm, and retention time of 1 hour. The temperature of the alloy flakes at the start of hydrogen absorption reaction was 25°C. Hydrogen desorption step—subsequent step—was performed under the conditions: vacuum of 13 Pa, 500°C, and retention time of 1 hour. Intermediate pulverization was performed by use of a Brawn mill, and the hydrogen-decrepitated powder was pulverized in a 100% nitrogen atmosphere to a particle size of 425  $\mu\text{m}$  or less. To the resultant powder, zinc stearate powder was added in an amount of 0.07% by mass. The mixture was sufficiently mixed in a 100% nitrogen atmosphere by use of a V-blender, and then micro-pulverized by use of a jet mill in a nitrogen atmosphere incorporated with oxygen (4,000 ppm), to a particle size of 3.2  $\mu\text{m}$  (FSSS). The resultant powder was sufficiently mixed again in a 100% nitrogen atmosphere by use of a V-blender. The obtained powder was found to have an oxygen concentration of 2,500 ppm. Through analysis of the carbon concentration of the powder, the zinc stearate content of the powder was calculated to be 0.05% by mass.

Subsequently, the thus-obtained powder was press-molded in a 100% nitrogen atmosphere and a lateral magnetic field by use of a molding apparatus. The molding pressure was 1.2 t/cm<sup>2</sup>, and the magnetic field in the mold cavity was controlled to 15 kOe.

The thus-obtained compact was maintained sequentially in vacuum of  $1.3 \times 10^{-3}$  Pa at 500°C for one hour and in vacuum of  $1.3 \times 10^{-3}$  Pa at 800°C for two hours, to thereby remove zinc stearate and hydrogen, and was further maintained in vacuum of  $1.3 \times 10^{-3}$  Pa at 1,060°C for two hours for sintering. The density of the sintered product was as sufficiently high as 7.52 g/cm<sup>3</sup>. The sintered product was further heat-treated at 540°C for one hour in an argon atmosphere.

Magnet characteristics of the sintered product as measured by means of a direct-current BH curve tracer were as follows: Br  $\approx$  13.9 kG; iHc = 10.6 kOe; and (BH)<sub>max</sub> = 45.4 MGOe. The sintered product was found to have an oxygen concentration of 3,100 ppm.

A cross-section of the sintered product was mirror-polished, and the polished surface was observed under a polarizing microscope. The obtained mean crystal grain size was 15-20  $\mu\text{m}$ , with substantial homogeneity in size.

#### Comparative Example 2

The alloy flakes produced in Comparative Example 1 were pulverized in a manner similar to that of Example 8, to thereby produce a powder having a particle size of 3.3  $\mu\text{m}$  (FSSS). The powder was found to have an oxygen concentration of 2,600 ppm. In a manner similar to that of Example 8, the powder was molded in a magnetic field and sintered, to thereby produce an anisotropic magnet. When the sintering temperature was 1,060°C, the density of the sintered product was 7.38 g/cm<sup>3</sup>, indicating insufficient sintering. Therefore, the sintering temperature was elevated to 1,090°C.

Magnet characteristics of the obtained product as measured by means of the same direct-current BH curve tracer as employed in Example 8 were as follows: Br = 13.5 kG; iHc = 9.8 kOe; and (BH)<sub>max</sub> = 42.8 MGOe, when sintering temperature was 1,060°C and Br = 13.8 kG; iHc = 7.4 kOe; and (BH)<sub>max</sub> = 35.2 MGOe, when sintering temperature was 1,090°C. These sintered products were found to have an oxygen concentration of 3,100 ppm and 3,200 ppm, respectively.

A cross-section of each sintered product was mirror-polished, and the polished surface was observed under a polarizing microscope. The mean crystal grain size of the product sintered at 1,060°C was 15-20  $\mu\text{m}$ , with substantial homogeneity in size. However, the product sintered at 1,090°C was predominantly formed of grains of approximately same grain size and had a mean crystal grain size of 20-25  $\mu\text{m}$ . In addition, crystal grains grown to a size of some tens to some hundreds  $\mu\text{m}$  were found in a number of sites.

In order to investigate the reason why a sufficient density could not be attained in the product sintered at 1,060°C, cross-sections of micro-pulverized particles were observed under a scanning electron microscope as electron-reflection images. Based on these images, the product of Example 8 was found to contain a number of grains having R-rich phase at their edges, whereas the product of Comparative Example 2 was found to contain a considerably small number of such grains and remarkably to contain grains formed exclusively of R-rich phase. Thus, the results indicated that the powder of

Comparative Example 2 had poor dispersibility of R-rich phase and failed to provide a sintered product having a sufficient density, when the powder was sintered at a sintering temperature similar to that employed in Example 8.

## CLAIMS

1. A method for controlling the metallographic structure of an alloy containing a rare earth element, which comprises the steps of:

melting said alloy containing a rare earth element in vacuum or under an inert gas;

pouring a molten metal obtained by melting said alloy onto a rotatable roll which is rotating and being cooled, to thereby cool the molten metal for solidification, said pouring being performed in a vacuum chamber or a chamber purged by an inert gas;

forming a strip of said metal through solidification;

immediately after solidification, reducing said resultant solidified strip into flakes;

collecting said resultant fragmented alloy flakes in a receptacle; and

controlling the cooling rate of said fragmented alloy flakes by means of a cooling medium.

2. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein said receptacle has a cooling partition and a gas or liquid serving as a cooling medium is allowed to pass through said cooling partition, to thereby control the cooling rate of said fragmented alloy flakes.

3. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein said receptacle is capable of controlling the cooling rate of said fragmented alloy flakes when an inert gas serving as a cooling medium is allowed to pass through the interior of said receptacle.

4. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein said inert gas is caused to flow through said cooling partition provided in said receptacle and having a vent.

5. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein said receptacle has a separation partition for controlling the cooling rate of said fragmented alloy flakes.

6. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, further comprising a step in which, subsequent to controlling of the cooling rate of the fragmented alloy flakes, transferring said fragmented alloy flakes from said chamber to another chamber for cooling said fragmented alloy flakes.
7. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein a strip casting method is employed for pouring said molten alloy containing a rare earth element onto said rotatable roll which is rotating, to thereby cool said molten alloy and form a strip through solidification.
8. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein the cooling rate of said fragmented alloy flakes is controlled, to thereby control average inter R-rich phase spacing to 3-15  $\mu\text{m}$ .
9. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein the average cooling rate of said fragmented alloy flakes within a range of 800 to 600°C is 10-300°C/min.
10. A method for controlling the metallographic structure of an alloy containing a rare earth element according to claim 1, wherein said alloy containing a rare earth element is an R-T-B alloy, wherein R represents at least one element selected from the group consisting of Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu; and T represents a substance predominantly comprising Fe, with a portion of Fe atoms being optionally substituted by Co, Ni, etc.
11. A powder of an alloy containing a rare earth element, which powder is produced by pulverizing fragmented alloy flakes obtained by a method according to claim 1 and having a thickness of 0.1-0.6 mm and an inter R-rich phase spacing of 3-15  $\mu\text{m}$ .
12. A magnet produced by shaping said powder of an alloy according to claim 11 and sintering the shaped product.

Fig. 1

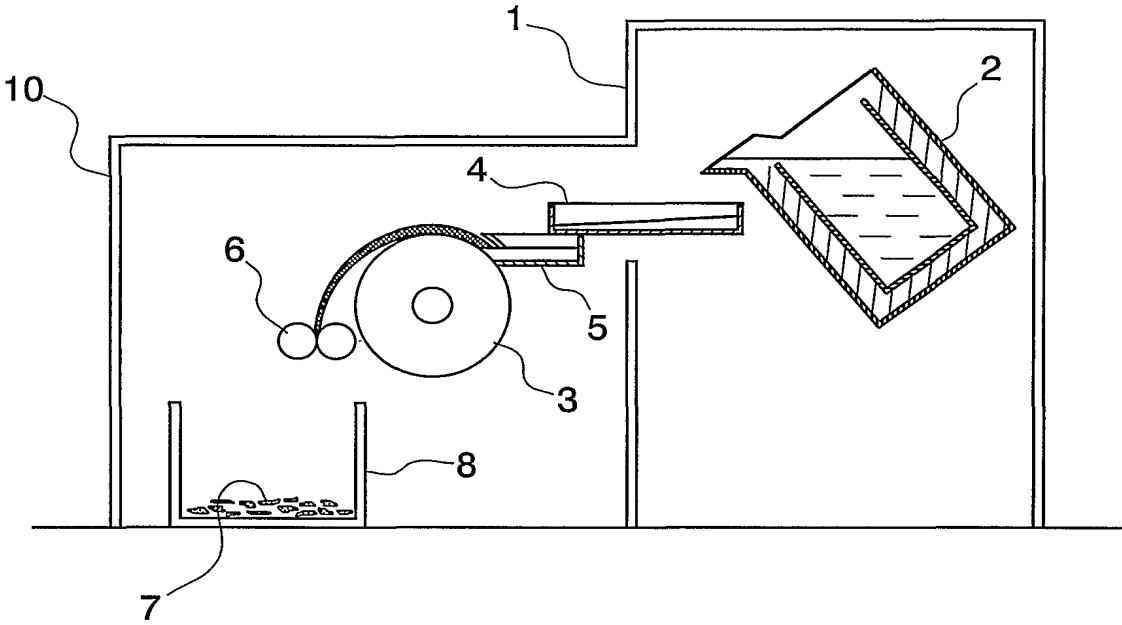


Fig. 2

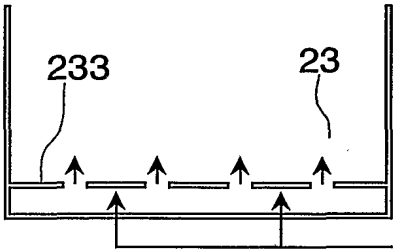
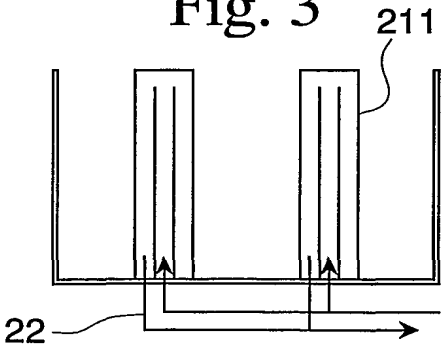


Fig. 3



2/2

Fig. 4

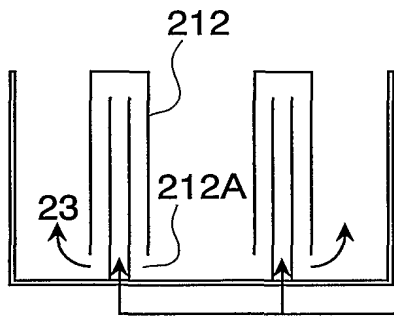


Fig. 5

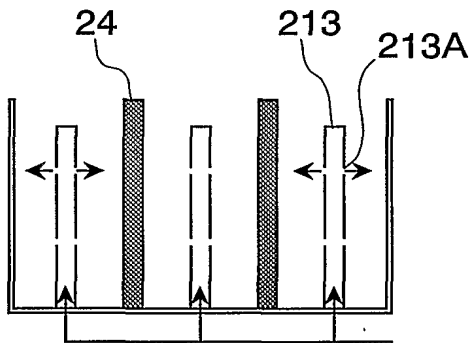


Fig. 6

